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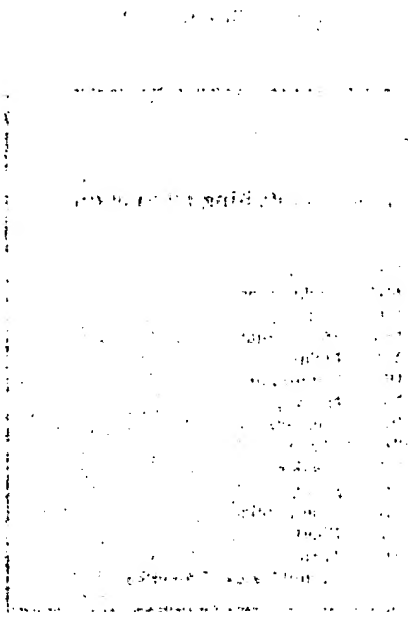
(54) Title: ARTIFICIAL JOJOBA OIL

(57) Abstract

A compound, the properties of which in many respects are analogous to corresponding properties of naturally occurring jojoba oil, is synthesized by sujecting material containing a precursor of the compound to a transesterification reaction to yield the compound. Preferably, the compound is synthesized by transesterifying erucic acid with 2-octyldodecanol in the presence, as catalyst, of concentrated sulphuric acid.



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ARTIFICIAL JOJOBA OIL

Technical Field

This invention relates generally to an artificially synthesized compound, the properties of which are analogous to certain of the properties of naturally occurring jojoba oil, and to a method of producing the artificially synthesized compound.

Background of the Invention

Although the present invention will be described with particular reference to artificially synthesized jojoba oil and to a method of producing this material it is to be noted that the scope of the present invention is not so limited but may extend to include other similar materials and methods.

Natural jojoba oil has been known for centuries for its lubricant and moisturising properties, and is of a neutral pH. Previous attempts have been made to produce artifical jojoba oil due to the high cost and problems associated with the availability of the natural product. However, such attempts have not been entirely satisfactory.

The Wicken Chemical Company of U.S.A., produced a product under the name of WICKENOL 139 which is styled along the lines of a Synthetic jojoba oil. It is believed that WICKENOL 139 is an ester formed from unsaturated straight chain acids, predominantly C_{22}-containing acids and branched chain alcohols, predominantly C_{20}-containing acids and thus, has the empirical formula $C_{12}H_{20}O_2$. WICKENOL 139 is characterized by the infra-red spectrograph as shown in figure 3.

Scher Chemicals Inc describe the production of an oil (known as SCHEROBA OIL) in GB 2106507 which the applicants believe to be an ester comprising predominantly isostearylerucyl enucate. GB 2106507 discloses the use of alcohols with odd numbers of carbon atoms. SCHEROBA OIL is characterized by the infra-red spectrograph as shown in figure 4.

The aforementioned artifical oils are not entir ly satisfactory. For example, the lubricity does not equal that of the naturally occurring jojoba oil.

Jojoba oil, both naturally occurring and artificially derived has many known commercial uses, which include such uses as high temperature lubricants, additives to mineral oil to improve their lubricity, additives and/or bases for cosmetic and skin care products such as moisturises, use in electronic components, slip agents in plastic films, leather softening agents, anti-foam agents in preparations of penicillin and tetracyclin, non-corrosive permanent lubricants for scientific instruments and robotics and the like. Naturally occurring jojoba oil is composed of esters of monounsaturated straight-chain alcohols and acids.

Following intensive research, the present inventors have found a method for preparing a compound, the properties of which in many respects are analogous to the corresponding properties of a naturally occurring jojoba oil. One preferred method involves the use of mustard seeds having a high erucic acid content, from which mustard seed oil is extracted and transesterified. Mustard seed crops are readily grown in Australia and elsewhere in the world and yield good quantities of mustard seed oil (typically one tonne per hectare). Typically, the oil yield from mustard seed which is available for processing is in the order of 35% by weight.

Summary of the Invention

In a first aspect, the present invention provides a method of producing an artificially synthesized compound, the properties of which in many respects are analogous to corresponding properties of naturally occurring jojoba oil, the method comprising subjecting material containing a precursor of the said compound to a transesterification reaction and yielding the said compound.

Preferably, the precursor is erucic acid, an erucic acid containing material, or a mixture thereof.

It is further preferred that the alcohol is a branched-chain alcohol, more preferably, 2-octyldodecanol (C_{20}) which is marketed by Henckel Chemicals under the trade name Eutanol G, stearyl alcohol (C_{18}), cetyl alcohol (C_{14}), 1-eicosanol (C_{20}), 2-eicosanol (C_{20}), oleyl alcohol, isomers thereof, or a mixture of one or more thereof.

It is further preferred that the material containing the percursor comprises material obtained from vegetable oils containing at least 2-66% erucic acid residues, mustard (Brassica) species, rape (high-erucic) species, Crambe abyssinica, or naturally occurring jojoba. Typically, the erucic acid is combined with other acids. More typically the acid reactant of the transesterification reaction is a combination or collection of all of the acids present in the parent mustard seed oil or mustard oil. However, it is to be noted that any suitable alcohol, acid or catalyst may be used in the practice of the present invention. Furthermore, it is to be noted that sources other than mustard seed may be used to obtain artificially derived jojoba oil and such sources include seed of rapeseed varieties whose oil is high in erucic acid content.

Preferred additional acids include oleic acid (C18:1), eicosenoic acid (C20:1), erucic acid (C22:1), nervonic acid (C24:1), linoleic acid (C18:2), or a mixture of one or more thereof.

Typically, the transesterification reaction mixture is heated for up to six hours at a temperature of up to 110°C and the resultant compound is extracted by a solvent. The solvent is preferably hexane or petroleum. The acid catalyst is typically a strong acid, such as sulphuric acid and is preferably concentrated sulphuric acid.

In a second aspect, the present invention provides an artificially synthesized compound, the properties of which in many respects are analogous to corresponding properties of naturally occurring jojoba oil.

Preferably, the artificially synthesized compound has the empirical formula $C_{40}H_{78}O_2$, and more pr ferably

the compound is characterized by having an infra-red spectrograph substantially as illustrated in figure 1.

Best Mode of Carrying out the Invention

Preferred embodiments of the present invention will now be described by way of Example only(which are not to limit the scope of the invention in any way) with reference to the accompanying drawings in which:

Figure 1 shows an infra-red spectrograph of one form of the compound of the present invention as described herein having the empirical formula $C_{40}H_{78}O_2$,

Figure 2 shows an infra-red spectrograph of refined natural jojoba oil,

Figure 3 shows an infra-red spectrograph of WICKENOL 139, and

Figure 4 shows an infra-red spectrograph of SHEROBA OIL.

Example 1

Mustard seed oil	25 grams
2-octyldodecanol	25 grams
concentrated H_2SO_4	1-2 grams

A reaction mixture is obtained by adding each of the above indicated reactants together in the proportion as indicated above. The reaction mixture is heated with stirring for approximately 2 hours at about 100°C in a water or oil bath. About 200 ml of water is added to the heated reaction mixture and the reaction mixture is then extracted from the water with hexane or petroleum ether in triplicate using 60 ml for each extraction (3 x 60 ml). The ether or hexane extracts are then combined into a single solution and are kept overnight over anhydrous Na_2SO_4 to remove any remaining traces of water. The solution is filtered and the solvent removed under reduced pressure to yield the ester. Typically, the amount of ester produced is about41.4 grams which corresponds to about 82% yield. The yellow colour of the product is optionally removed by passing the product through activated silica.

The material made by Example I was subjected to column chromatography and a sample of the eluted material taken. The infra-red spectrograph was obtained using this sample of the eluted material. The infra-red spectra of naturally occurring jojoba oil exhibits peaks at the following locations,

(i) a very strong triplet from 3000 to 2800,
(ii) a moderately strong peak at about 1740,
(iii) two weak single peaks at 1470 and 1170, respectively.

As can be readily seen from a comparison of Figures 1 and 2, the compound forming the subject matter of the present invention as evidenced by the infra-red spectra of Figure 1 has an infra-red spectra which mimics that obtained from naturally refined jojoba oil as is evidenced by the spectra shown in Figure 2, and thus may be regarded as an artificially derived synthetic substitute for naturally occurring jojoba oil.

The spectra of WICKENOL 139 shown in Figure 3 and that of SCHEROBA OIL shown in Figure 4 both contain additional peaks in the region 1400 to 1100 which illustrates that the compound produced by the present invention is more closely related to the naturally refined jojoba oil than is either WICKENOL 139 or SCHEROBA OIL.

Similar methods to those described for Example 1 were used to produce further synthetic substitutes for naturally occurring jojoba oil:

Example 2

A reaction mixture is obtained by adding each of the following reactants together:

High erucic acid rapeseed oil plus equal weight of oleyl alcohol (cis-9-octadecen-1-ol) with H_2SO_4 catalyst.

The product is an ester which is coloured, and has a detectable odour.

Example 3

A reaction mixture is obtained by adding each of the following reactants together:

High erucic acid rapeseed oil plus equal weight of stearyl alcohol (octadecon-1-ol) with H_2SO_4 catalyst.

The product is an ester which is solid at room temperature.

Example 4

A reaction mixture is obtained by adding each of the following reactants together:

High erucic acid rapeseed oil plus equal weight cetyl alcohol (hexadecan-1-ol) gives ester product which is a solid at room temperature.

High erucic mustard seed oil was used in the above trials with similar results.

Claims:-

1. A method of producing an artificially synthesized compound, the properties of which in many respects are analogous to corresponding properties of naturally occurring jojoba oil, the method comprising subjecting material containing a precursor of the compound to a transesterification reaction to yield the compound.

2. A method as claimed in claim 1 wherein the precursor is erucic acid, an erucic acid containing material, or a mixture thereof which is transesterified with an alcohol in the presence of an acid catalyst.

3. A method as claimed in claim 2 wherein the alcohol is a branched-chain alcohol, stearyl alcohol (C_{18}), cetyl alcohol (C_{14}), 1-eicosonol (C_{20}), 2-eicosanol (C_{20}), isomers thereof, or a mixture of one or more thereof.

4. A method as claimed in 3 wherein the alcohol is 2-octyl dodecanol or oleyl alcohol.

5. A method as claimed in any one of the preceding claims wherein the material containing the precursor comprises material obtained from one or more of the following sources:

vegetable oils containing at least 2-66% erucic acid residues, mustard (Brassica) species, rape (high-erucic) species, Crambe abyssinica or naturally occurring jojoba.

6. A method as claimed in claim 5 wherein the material containing the precursor further comprises oleic acid (C18:1), eicosenoic acid (C20:1), erucic acid (C22:1), nervonic acid (C24:1), linoleic acid (C18:2), or a mixture of one or more thereof.

7. A method as claimed in any one of claims 1-4 wherein the material containing the precursor is mustard seed oil or oil extracted from mustard seed.

8. A method as claimed in any one of the preceding claims wherein the transesterification reaction mixture is heated for up to six hours at a temperature of up to 110°C.

9. A method as claimed in any one of the preceding claims wherein the solvent for the transesterification reaction is water.

10. A method as claimed in any one of the preceding claims wherein the compound is extracted from the transesterification reaction mixture by a solvent.

11. A method as claimed in claim 10 wherein the solvent is hexane or petroleum.

12. A method as claimed in any one of the preceding claims wherein the acid catalyst is sulphuric acid.

13. A method as claimed in claim 12 wherein the sulphuric acid is concentrated sulphuric acid.

14. A method of producing an artificially synthesised compound, the properties of which in many respects are analogous to the corresponding properties of naturally occuring jojoba oil substantially as described herein with reference to any one of the Examples.

15. An artificially synthesized compound, the properties of which in many respects are analogous to the corresponding properties of naturally occurring jojoba oil, having subtantially the infra-red spectrograph as shown in Figure 1.

16. An artificially synthesised compound, the properties of which in many respects are analogous to the corresponding properties of naturally occuring jojoba oil, prepared by a method as claimed in any one of claims 1-14.

17. An artificially synthesised compound, the properties of which in many respects are analagous to the corresponding properties of naturally occurring jojoba oil substantially as described herein with reference to any one of the Examples.

18. An artificially synthesised compound as claimed in any one of claims 15-17 having the empirical formula $C_{40}H_{78}O_2$.

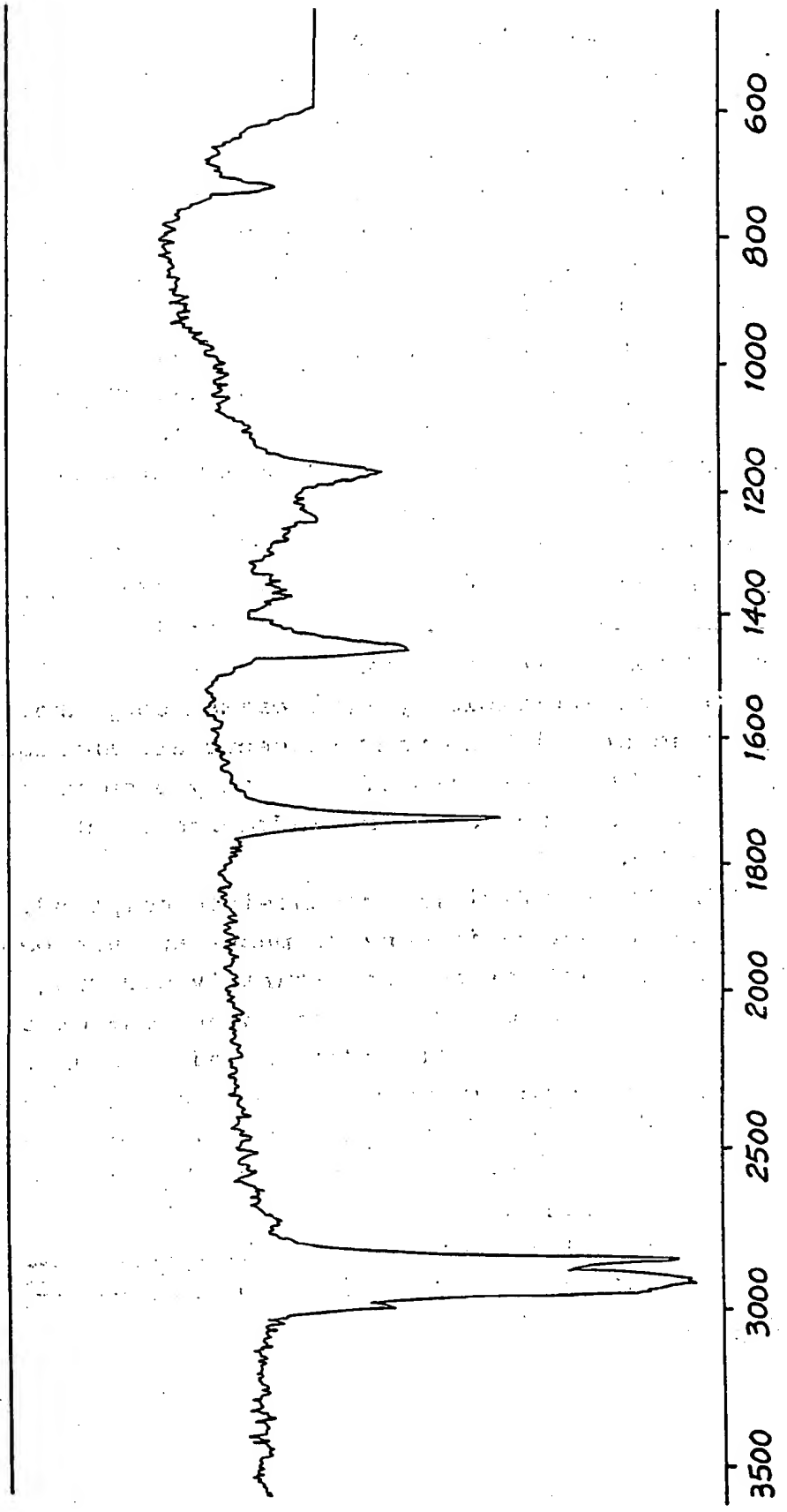

FIG. 1

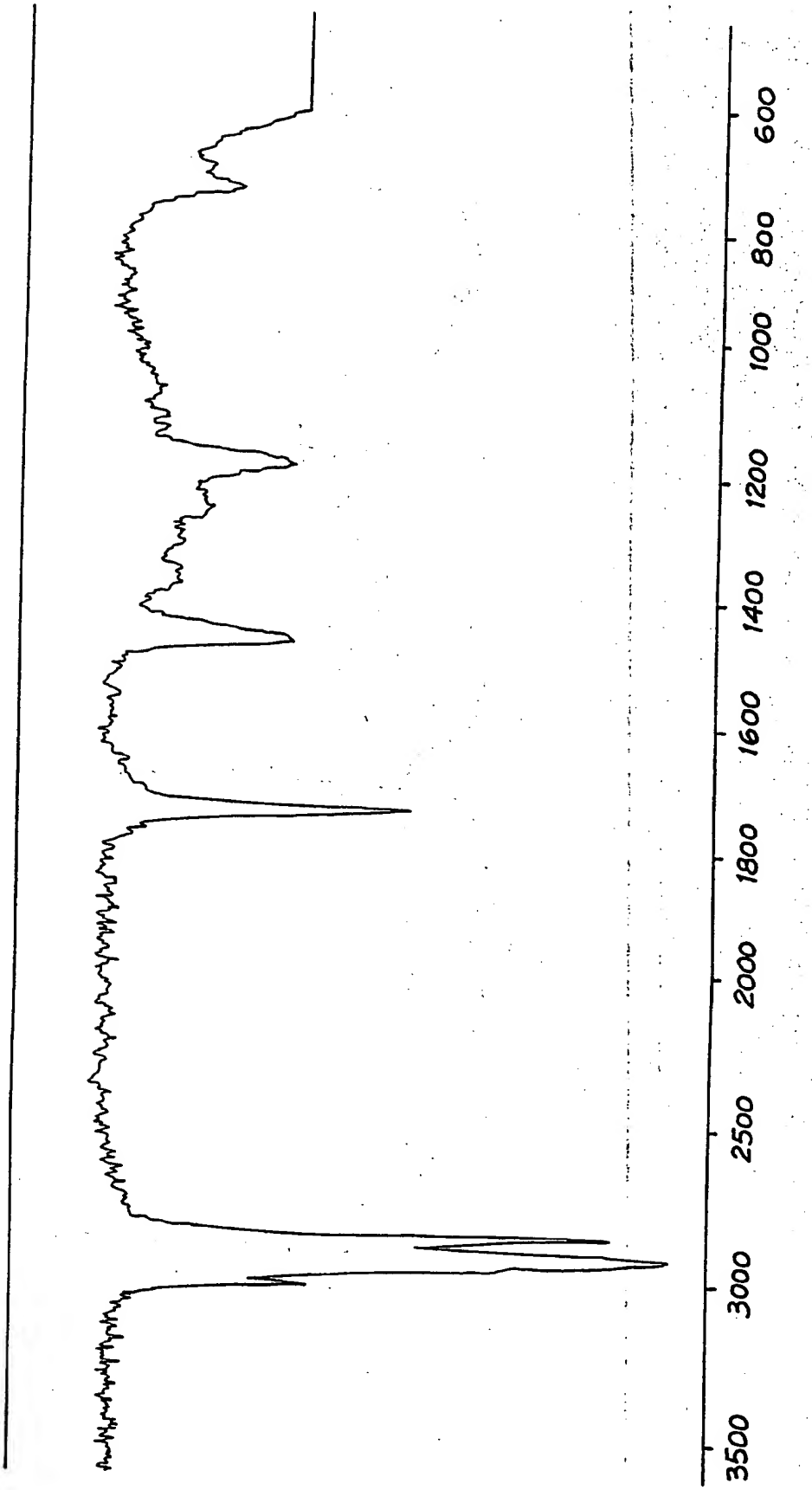

FIG. 2

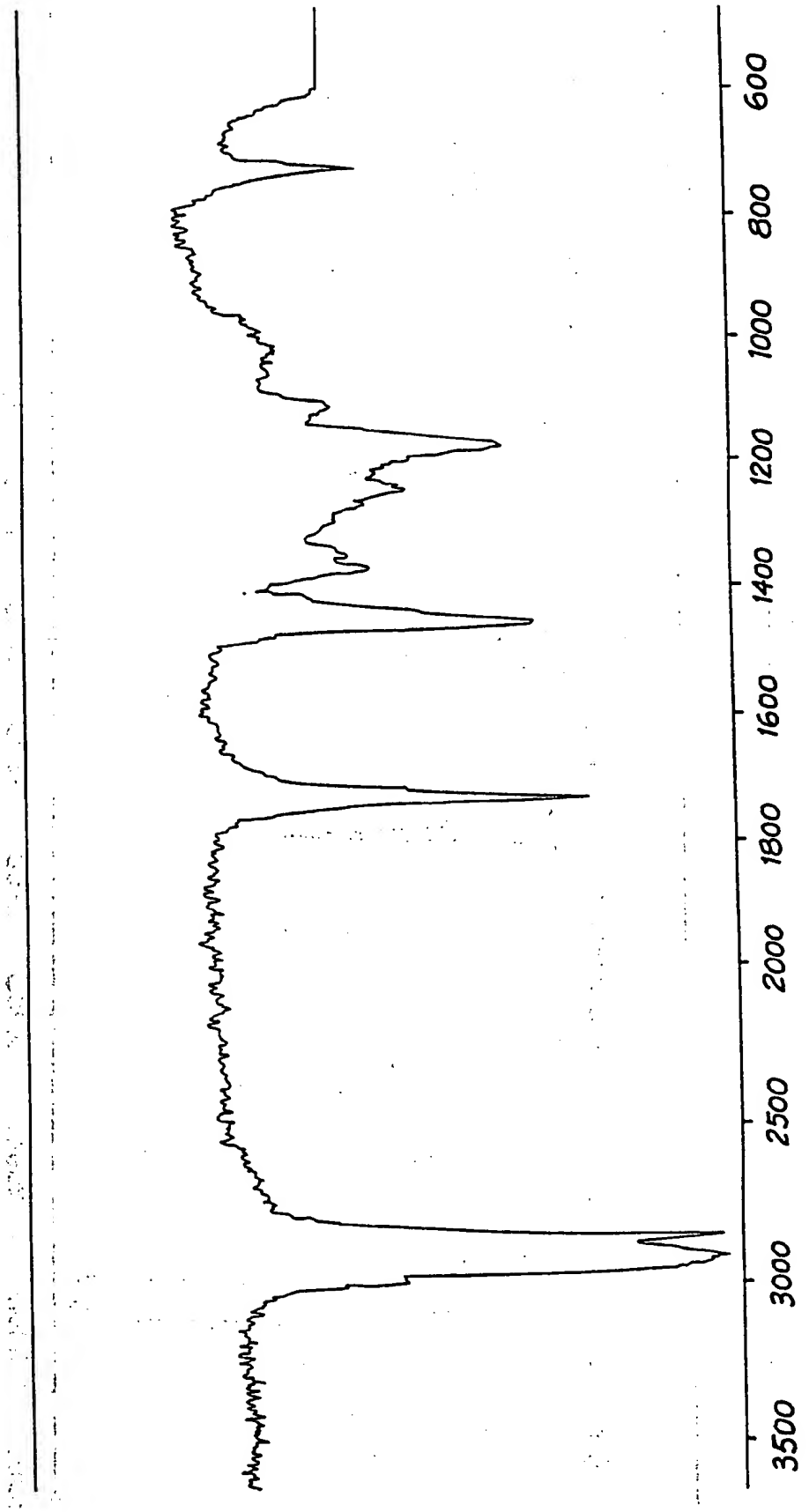

FIG. 3

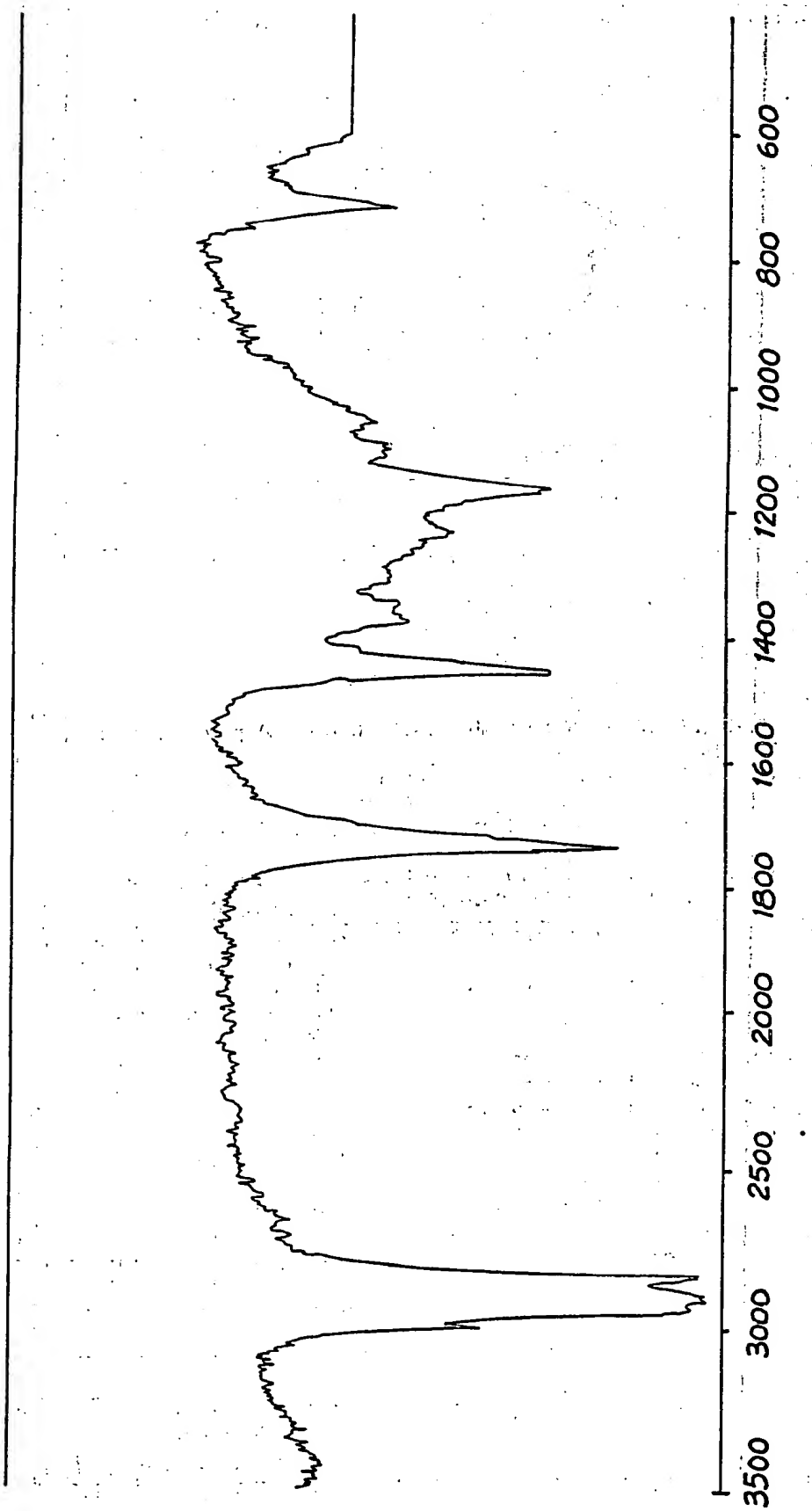

FIG. 4

INTERNATIONAL SEARCH REPORT

International Application No. PCT/AU 90/00253

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) 6

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl.[5] C11C 3/10, C07C 067/02, 069/533

II. FIELDS SEARCHED

Minimum Documentation Searched 7

Classification System	Classification Symbols
IPC[3-5]	C11C 3/10, C07C 067/02, 069/533
IPC[2]	C07C 069/52, 069/53

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched 8

III. DOCUMENTS CONSIDERED TO BE RELEVANT 9

Category*	Citation of Document, [11] with indication, [12] where appropriate, of the relevant passages	Relevant to Claim No 13
X	AU,B, 11275/83 (561896) (DYNAMIT NOBEL AG) 15 September 1983 (15.09.83) whole document	1, 2, 4, 10, 16-18
X	AU,A, 43950/72 (EMERY INDUSTRIES) 3 January 1974 (03.01.74) page 4 line 6-page 7 line 6	1-3, 5-6, 16
X	EP,A, 69946 (CIRTA) 19 January 1983 (19.01.83) whole document	1-7, 16-18
X	EP,A, 165457 (HENKEL KGaA) 27 December 1985 (27.12.85) whole document	1, 2, 16
X	EP,A, 195675 (KAO CORP) 24 September 1986, (24.09.86) page 4 line 5-page 6 line 14	1-4, 16, 17
X	Industrial and Engineering Chemistry Research, Volume 27, no. 11, 1988, pages 2179-2182, (Washington, USA) Martinez et al, "Synthesis of Esters of High Molecular Weight. An Analogue of Jojoba Oil. A Statistical Approach"	1, 16
X	Patent Abstracts of Japan, C-197, page 40, JP,A, 58-148836 (SHINEI KAGAKU K K) 5 September 1983 (05.09.83) (continued)	1-4, 16-17

* Special categories of cited documents: 10

"A" document defining the general state of the art which is not considered to be of particular relevance

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
7 September 1990 (07.09.90)	20 September 1990
International Searching Authority	**Signature of Authorized Officer**
Australian Patent Office	GORDON MASTERS Gordon Masters

Form PCT/ISA/210 (second sheet) (January 1985)

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

X,Y	GB,A, 2106507 (SCHER CHEMICALS INC) 13 April 1983 (13.04.83) whole document	1-7, 16-18
X,Y	Journal of Agricultural Food Chemistry Volume 36, 1988, pages 1333-1336, (Washington, USA), Mukherjee et al, "Preparation of Esters Resembling Natural Waxes by Lipase-Catalyzed Reactions"	1-7, 16

V.[] OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE 1

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. [] Claim numbers ..., because they relate to subject matter not required to be searched by this Authority, namely:

2. [] Claim numbers ..., because they relate to parts of the international application that do comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. [] Claim numbers ..., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4 (a):

VI.[] OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING 2

This International Searching Authority found multiple inventions in this international application as follows:

1. [] As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. [] As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. [] No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. [] As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

[] The additional search fees were accompanied by applicant's protest.
[] No protest accompanied the payment of additional search fees.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON
INTERNATIONAL APPLICATION NO. PCT/AU 90/253

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report	Patent Family Members		
AU 11275/83	BR 8301219	CA 1215072	DE 3208930
	DK 1181/83	EP 88895	ES 520491
	JP 58167543	US 4510093	
AU 43950/72	BE 785416	CA 1111017	DE 2232323
	FR 2144334	GB 1379105	IT 965846
	JP 48056706	US 3740333	
EP 69946	DE 3270747	EP 69946	FR 2509322
	JP 28065794		
EP 165457	DE 3418887	JP 60260508	
EP 195675	CA 1258008	DE 3664969	ES 553164
	US 4675250	JP 62001118	JP 62001119
GB 2106507	DE 3220973	FR 2513245	JP 58065248
JP 29804			

END OF ANNEX

[redacted]